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Assessment of Heavy Metals and PCB's at Selected Sludge Application Sites in Ontario

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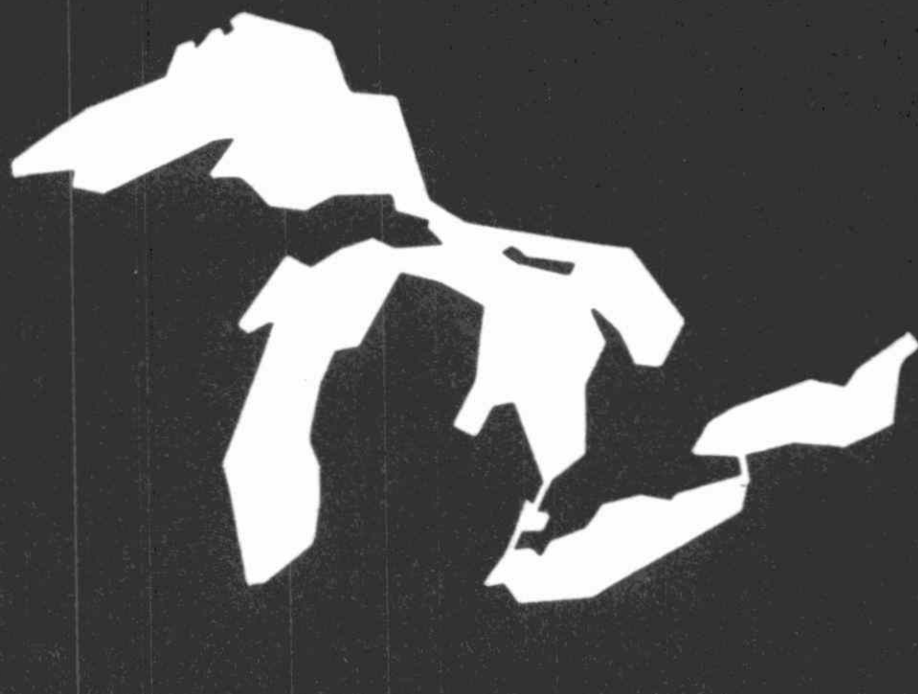

Research Program for the Abatement of Municipal Pollution under Provisions of the Canada-Ontario Agreement on Great Lakes Water Quality

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ASSESSMENT OF HEAVY METALS AND PCB'S AT SELECTED SLUDGE APPLICATION SITES IN ONTARIO

by

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ABSTRACT

Ten sludge applications sites in Ontario were sampled during the summers of 1976 and 1978. The sites were selected to represent different locations, soil types, sludging histories and metal and PCB concentrations in sludges. Concurrently, control (i.e., no sludge) sites adjacent to each sludge application site were sampled to determine the background levels of metals and PCB's in the soils.

Until recently, few records were kept of sludge application on land in Ontario. Consequently, it was necessary to estimate the metal and PCB loadings to the sites. It was estimated that metal loadings at several locations exceeded the maxima recommended in the Ontario Sludge Utilization Guidelines.

Sludge application increased the total metal and PCB contents of soils and the largest increases generally coincided with the largest estimated loadings. DTPA-extractable metals in the soils were also increased and were well correlated with total metals in the soils.

In general, sludge application exhibited small effects on plant composition. Frequently, the differences between sites were larger than the sludge treatment effects. The most consistent effects of sludge treatment were to increase the Cd and Zn contents of plant materials. However, with a few exceptions, the increases were small and not well related to estimated metal loadings in sludge or to total or DTPA-extractable Cd and Zn in the soils. There was no consistent effect of sludge on the Cu, Ni, Pb, Mo (except at Stratford), Mn, Cr, As, Hg and PCB contents of plant materials. Stratford sludge contributed a heavy Mo loading and greatly increased the Mo content of corn leaves at that site.

Increased concentrations of Zn in plant materials resulting from sludge application are desirable because Zn tends to be deficient for plant and animal nutrition. However, increased levels of Cd are undesirable because Cd has no known biological function and it is toxic to both plants and animals. An exclusive diet of corn leaves from the Stratford sludge application site would cause molybdenosis (molybdenum-induced copper deficiency) in ruminant animals.

Results indicated that sludge application to land according to the Ontario Guidelines will not cause deleterious uptake of heavy metals by corn, oats and wheat. It appeared that the Guidelines allow an appreciable safety margin. This is desirable since sludge compositions vary widely with time.

RÉSUMÉ

Au cours de l'été de 1976 et de 1978, on a utilisé comme échantillon dix champs d'épandage de boues situés en Ontario. Ces endroits différaient par leur emplacement, le type de leur sol, les épandages qui y avaient été effectuées et les concentrations de métaux et de BPC dans les boues répandues. En même temps, on a utilisé comme groupe témoin des endroits ne servant pas à l'épandage des boues et contigus à chaque champ d'épandage pour y déterminer les concentrations naturelles de métaux et de BPC dans le sol.

Jusqu'à récemment, on ne recueillait que peu de reseignements sur l'épandage des boues en Ontario. Il a donc fallu estimer la valeur des apports de métaux et de BPC en ces endroits. On a jugé que dans plusieurs lieux, l'apport de métaux dépassait les cotes maximales indiquées dans les directives ontariennes sur l'utilisation des boues.

Les épandages avaient eu pour effet d'accroître la teneur en métaux totaux et en BPC des sols; en général, on observait les augmentations les plus importantes en des endroits où les épandages avaient été probablement les plus volumineux. La teneur en métaux extractibles par le DTPA avait aussi augmenté et elle était dans une bonne mesure proportionnelle à la teneur en métaux totaux dans les sols.

En général, les épandages ont eu des effets minimes sur la composition des plantes. Fréquemment, les différences à ce point de vue entre les endroits de l'échantillon étaient plus remarquables que les effets des épandages. Les épandages ont eu surtout pour effet d'augmenter la teneur en Cd et en Zn des végétaux. Sauf un petit nombre d'exceptions, cette augmentation était modeste et correspondait peu à l'apport en métaux provenant des boues que l'on avait évalué ou à la teneur en Cd et en Zn totaux ou extractibles par le DTPA, dans les sols. Les épandages n'ont pas eu non plus d'effets constants sur la teneur en Cu, en Ni, en Pb, en Mo (sauf à Stratford) en Mn, en Cr, en As, en Hg et en BPC des végétaux. À Stratford, les boues contenaient beaucoup de Mo et la teneur en Mo des feuilles de maïs a beaucoup augmenté.

L'augmentation de la teneur en Zn des végétaux est valable parce que cet élément tend à manquer dans l'alimentation des végétaux et des

animaux. D'autre part, l'augmentation de la teneur en Cd n'est pas souhaitable parce que cet élément n'a aucune fonction biologique connue et il est toxique pour les plantes et les animaux. Les ruminants qui se nourriraient exclusivement de feuilles de maïs du champ d'épandage de Stratford contracteraient une molybdénose (résultat d'une carence en cuivre provoquée par le molybdène).

L'étude laisse entendre en conclusion que si l'épandage de boues se conforme aux directives ontariennes, il n'y aura pas d'accumulation de métaux lourds dans le maïs, l'avoine et le blé. Il semble que ces directives assurent une marge de sécurité appréciable, d'autant plus souhaitable que la composition des boues varie beaucoup avec le temps.

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CONCLUSIONS

1. Estimates of sludge loadings to land, based on recall by the Water Pollution Control Plant operators and farmers were approximate at best.
2. Metal loadings to several sludge application sites probably exceeded the maxima recommended in the Ontario Sludge Utilization Guidelines.
3. Sludge treatment increased the total Cd, Zn, Cu, Ni, Pb, Hg and PCB contents of soils.
4. Sludge treatment increased the DTPA-extractable Cd, Zn, Cu, Ni and Pb contents of soils.
5. Sludge treatment increased the Cd, Zn and, at Stratford, the Mo contents of plant materials but exhibited no consistent effect on the Cu, Ni, Pb, Mn, Cr, As, Hg and PCB contents of plant materials.
6. The metal contents of crops grown on the sludge treated soils were within suggested normal ranges with one exception. Corn grown at Stratford exhibited an unacceptably high Mo content and would cause molybdenosis (Mo-induced Cu deficiency) when fed to cattle or sheep receiving no Cu supplement and little other forage.
7. Sludge application to land according to the Ontario Guidelines will not cause deleterious uptake of heavy metals by corn, oats or wheat. There appears to be an appreciable safety margin associated with the Guidelines.

1 INTRODUCTION

The major sinks available for wastewater sludge disposal are oceans, the atmosphere and land, and choice of a disposal technique requires evaluation of the benefits and risks associated with each. Ember (1975) reported that the risks of ocean disposal outweigh the potential benefits because aquatic organisms tend to accumulate metals and other toxic constituents in extremely high concentrations.

Incineration is the major disposal method employed in Ontario and it accounts for 40% (62 000 tonnes/a) of all sludge generated in the province (Antonic et al, 1980). However, it may require a large energy input and at the 700 to 900°C temperature levels generally used for sludge incineration, substantial amounts of cadmium, arsenic and mercury volatilize (Schroeder et al, 1981). The degree of loss of these elements to the atmosphere and contamination of the environment in the "fall-out" zone depends upon scrubber efficiency.

Application of sewage sludge to farmland during 1975 accounted for 34% (53 000 tonnes) of all sludge generated in Ontario (Antonic et al, 1980). The benefits of this disposal method to Water Pollution Control plants are the relative simplicity and low cost compared to other methods. The benefits to farmers are the soil conditioning material and plant nutrients supplied at no cost. Land application of sludge involves no risk for the treatment plants, however, there are potential short and long-term risks for the soil environment.

Nitrogen and pathogens in sludges represent short-term risks for the soil environment and must be considered when determining annual application rates and the crops to be grown. Toxic metals and organic compounds such as PCB's represent long-term risks because they accumulate in soils and may persist indefinitely. High levels of cadmium, zinc, copper, nickel, cobalt and boron in soils are toxic to plants and can severely reduce crop yields. Moreover, levels of cadmium and copper in plants which are too low to reduce yields, may be high enough to be toxic to animals. PCB's are chemically similar to DDT and are readily incorporated into animal tissue. No practical technology has been developed to reduce the levels of heavy metals and PCB's in soils. Thus, the only way to protect soil quality for future agricultural production is to establish maximum acceptable levels of these constituents in soil and to ensure that they are not exceeded.

No information was available concerning the levels of heavy metals and PCB's in Ontario agricultural soils receiving sludge and in crops grown on these soils. Consequently, a study sponsored by the Canada-Ontario Agreement on Great Lakes Water Quality was undertaken. The objective of the study was to determine whether land application of sludge has increased the levels of heavy metals and PCB's in Ontario soils and crops.

2 METHODS AND MATERIALS

2.1 Sludge Application Sites

Ten sludge application sites were sampled during the summers of 1976 and 1978. The sites were selected to represent:

1) Different locations within Ontario (Figure 1).
2) A range of soil types (Table 1).
3) A range of sludging histories (Table 1).
4) A range of heavy metal and PCB concentrations in sludges (Table 2).

To determine background heavy metal and PCB concentrations in the soils, control (i.e., no sludge) sites adjacent to the sludge application sites were sampled

In general, samples were taken from the same sites in 1976 and 1978. However, in 1978 some of the sites supported no crop or a different crop than in 1976. Where this occurred an effort was made to obtain samples from a different site, on the same farm or an adjacent farm, that was growing the same crop as in 1976. The samples obtained from different sites in 1976 and 1978 are identified in Table 1.

2.2 Sludges

Grab samples of the liquid sludges applied at each of the ten sites were taken during 1976. Three replicate samples of sludge were taken during one day from each Water Pollution Control Plant. Monteith and Stephenson (1978) estimated that, using this sampling technique, mean parameter concentrations in a given batch of sludge can be estimated within the range ±7 to ±55%. The sludges were oven-dried at 60°C, ground in a Wiley mill to pass a 60-mesh stainless steel screen and analyzed.

2.3 Soils

Soil samples from the sludge application sites and nearby control sites were collected during 1976 and 1978. Cores to a depth of 15 cm were taken using a 2 cm diameter sleeve sampler. Twenty such cores, taken at 20 m intervals on a 60 by 80 m grid, were combined to form one composite sample (Figure 2). Composite samples were taken from each of four areas spaced at

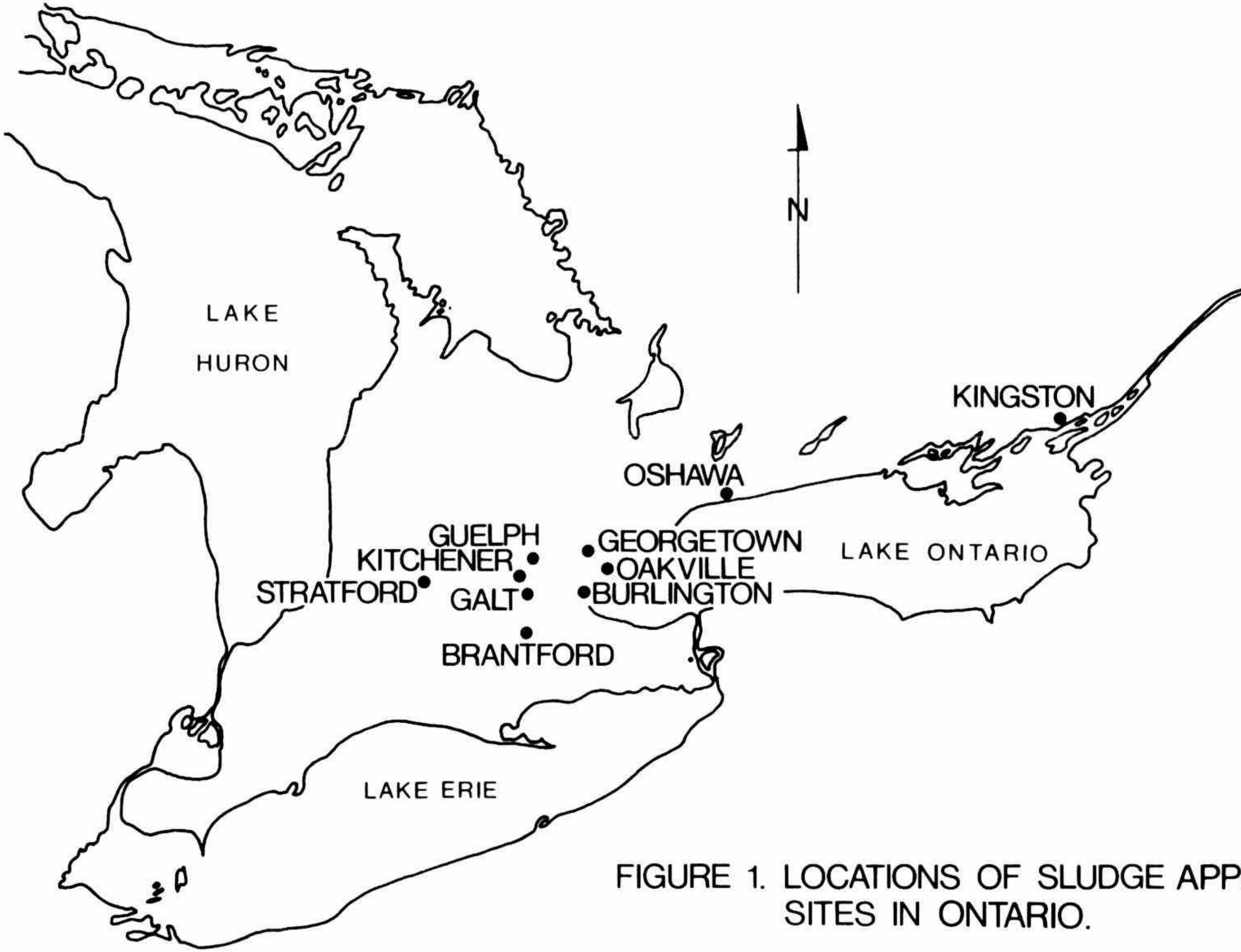

FIGURE 1. LOCATIONS OF SLUDGE APPLICATION SITES IN ONTARIO.

TABLE 1. SITE INFORMATION

Site		Soil Series	Texture	Drainage	Soil pH*		Approx. Years Sludge Applied	Sludge Applications/a	Approx. Annual Rate of Sludge Solids** Metric t/ha (tons/acre)
					Control site	Sludge site			
Brantford		Fairchild***	Loam	Good	7.1	7.0	4	3	4.5 (2)
Burlington	1976	Oneida, Jeddo	Clay loam	Good to	NM	NM	10	2	4.5 (2)
	1978	and Chinguacousy		poor	6.3	5.8	2	1	4.5 (2)
Galt		Dumphries, Mannheim and Tuscola	Loam	Good to imperfect	6.6	6.1	10	1	4.5 (2)
Georgetown		Oneida and Chinguacousy	Clay loam	Good to imperfect	6.1	5.7	4	1	4.5 (2)
Guelph	1976	Fox and Granby	Sandy loam	Good	NM	NM	4	1	4.5 (2)
	1978				6.2	6.3	2	1	4.5 (2)
Kingston	1976	White Lake and	Gravelly	Good	NM	NM	4	1	20 (9)
	1978	Uplands	sandy loam and sandy loam		4.8	6.0	1	1	Large
Kitchener		Caledon and Burford	Sandy loam and gravelly loam	Good	6.6	6.4	10	2	4.5 (2)
Oakville	1976	Oneida, Jeddo	Clay loam	Good to	NM	NM	8	1	7.6 (3.4)
	1978	and Chinguacousy		poor	5.6	5.9	15	1	4.5 (2)
Oshawa		Darlington	Clay loam	Good	6.8	6.6	10	1	1.0 (0.4)
Stratford		Perth and Brookston	Silt loam	Imperfect to poor	6.5	6.5	21	1	4.5 (2)

* Measured in 0.01 M $CaCl_2$.
** Average sludge application rate for Ontario 4.5 tonnes/ha (2 tons/acre) (Antonic et al, 1980).
*** Unpublished soil survey information for Brant County.
NM Not measured.

TABLE 2. SELECTED PROPERTIES OF SEWAGE SLUDGES* APPLIED AT TEN AGRICULTURAL SITES IN ONTARIO

Sludge Source	Concentrations (μg/g dry weight)										
	Cd	Zn	Cu	Ni	Pb	Cr	Mn	Mo	As	Hg	PCB's
Brantford	23	2200	1240	199	1230	697	244	<20	16.1	3.1	0.528
Burlington (Skyway)	214	5060	1360	403	1240	1780	299	<20	36.1	4.0	1.61
Galt	54	1490	1180	53	305	1270	264	<20	9.7	5.5	0.156
Georgetown	47	646	556	51	1190	316	183	<20	3.9	2.4	0.279
Guelph	206	7220	3680	88	1440	3910	271	64	13.1	15.6	0.715
Kingston	7.5	1780	626	26	893	1380	270	32	7.2	10.5	0.821
Kitchener	12.3	6610	658	333	615	6980	252	<20	9.9	3.1	0.651
Oakville (Southwest)	45	6820	1510	338	1350	2010	285	<20	7.3	8.0	0.396
Oshawa (Harmony Creek)	7.3	6100	683	1120	1340	6560	322	63	19.8	3.3	0.130
Stratford	33	2740	1180	1260	956	1540	471	206	17.7	3.4	0.227

* All sludges were secondary digested except for Burlington which was waste activated.

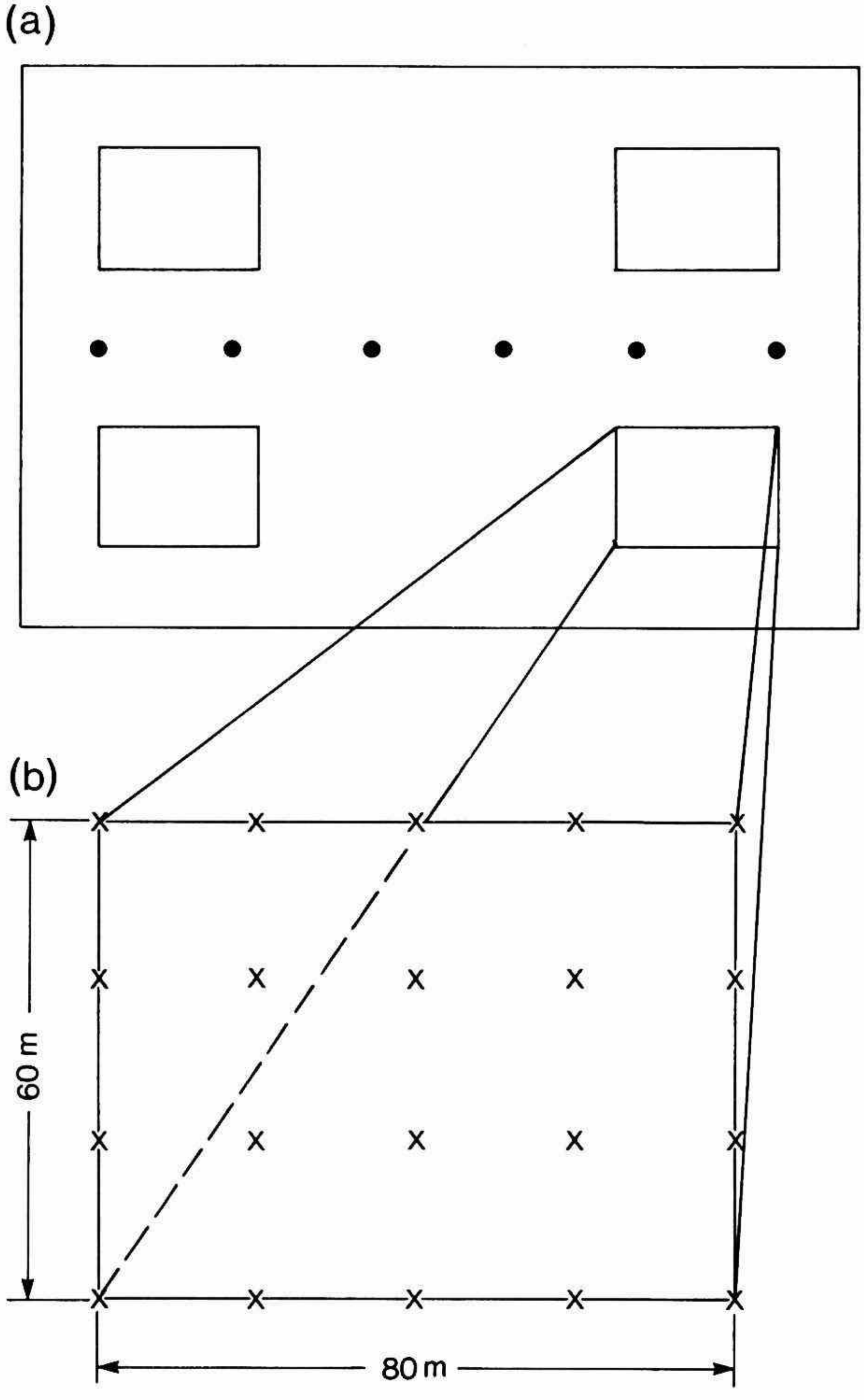

FIGURE 2. Soil and plant material sampling pattern in the field.

regular intervals within each site. In addition, grab samples of soil were taken from two sludge treated and two control sites in 1978. Each grab sample consisted of several cores from an area of about one square meter. Six grab samples were taken at approximately equidistant intervals along the length of the fields. The soils were air-dried and ground with a mortar and pestle to pass a 2 mm nylon screen. Approximately 10 g subsamples of the 2 mm soil were ground to pass a 300-mesh screen and were employed for the total metal analyses.

2.4 Plant Materials

In 1976, seven of the study sites were cropped to corn (*Zea mays* L), two were cropped to oats (*Avena sativa* L) and one was cropped to wheat (*Triticum aestivum* L). In 1978, eight of the study sites were cropped to corn, one was cropped to oats and one was not cropped. The plant materials were sampled concurrently with the soils. The second, third and fourth leaves from the top of corn plants were collected following tasseling and the above ground portions of oats and wheat were collected following heading out. Twenty subsamples of plant material taken at 20 m intervals on a 60 by 80 m grid were combined to form one composite sample (Figure 2). Composite samples were taken from each of four areas spaced at regular intervals within each of the sludge application and nearby control sites. In addition, grab samples of plant material were taken at the soil grab sample locations in 1978. The samples were oven-dried at 70°C and ground with a Wiley mill to pass a 60-mesh stainless steel screen. In 1978 a composite of the leaves, stems and heads of oats were ground and analyzed, whereas in 1976 a composite of only the leaves and stems of oats and wheat were ground and analyzed.

2.5 Analytical

Total metals in the sludge and plant materials were determined according to procedures outlined in the Analytical Methods Manual of the Wastewater Technology Centre, Burlington, Ontario. In brief, the sludges were extracted with hot aqua regia. The plant materials were dry-ashed overnight at 450°C and the ash digested in aqua regia, hydrogen peroxide and hydrofluoric acid. Arsenic in the sludges, plant materials and soils was determined

following potassium hydroxide-magnesium oxide fusion, and mercury was determined following sulphuric-nitric acid digestion. Total cadmium, zinc, copper, nickel, lead and molybdenum in the soils were determined at the Land Resource Research Institute, Ottawa following complete digestion with hydrogen peroxide and a combination of nitric, perchloric and hydrofluoric acids (McKeague, 1976). Values for total chromium in the soils were not reproducible by this technique and have not been reported. The 0.005 M DTPA-extractable metals in the soils were determined, also at the Land Resource Research Institute according to the method recommended by Lindsay and Norvell (1978). Total polychlorinated biphenyls (PCB's) in the sludges, plant materials and soils were determined following extraction into organic solvents. Measurements on the 1976 samples were done at the Ontario Ministry of Agriculture and Food, Pesticide Residue Testing Laboratory, Guelph, Ontario, and on the 1978 samples were done at the Wastewater Technology Centre, Burlington, Ontario.

Cadmium (Cd), zinc (Zn), copper (Cu), nickel (Ni), lead (Pb), molybdenum (Mo), manganese (Mn) and chromium (Cr) in the digests and extracts were measured by flame atomic absorption spectrometry. Arsenic (As) was measured colorimetrically and mercury (Hg) was measured by flameless atomic absorption. Total PCB's in the organic extracts were measured by gas chromatography.

3 RESULTS AND DISCUSSION

3.1 Sludge Application Sites

The same sludge application sites were sampled in 1976 and 1978 at all locations except Burlington, Guelph, Kingston and Oakville (Table 1). At Burlington, Guelph and Oakville, the sludge sites sampled in 1978 were on the same farms as those sampled in 1976 and at Kingston the sludge site was on an adjacent farm. The soils exhibited a wide range of textures and drainage characteristics. They were slightly to moderately acid except for the Brantford site where soil pH was 7.0.

In general, no record has been kept of sludge application on agricultural land in Ontario. (However, record systems were established during 1979-80 as part of the phasing-in program for the Sludge Use Guidelines). Few of the Water Pollution Control Plant superintendents that were contacted during this study knew where sludge had been spread and none knew the rates of application. The local farmers knew where sludge had been applied and in most cases recalled an approximate number of applications to their own land, however, few could estimate the application rates. Consequently, the sludge application records shown in Table 1 are approximate and in many instances the rate was assumed to be the 4.5 tonnes/ha (2 tons/acre) average rate estimated by Antonic et al (1980).

3.2 Sludges

Grab samples of the sludges applied at the study sites exhibited wide ranges of metal concentrations (Table 2) typical of Ontario sludges (Antonic et al, 1980; Monteith and Stephenson, 1978). All except the Georgetown sludge contained large amounts of one or more metals, probably derived from industrial sources. The Burlington and Guelph sludges exhibited large amounts of Cd; Oshawa and Stratford sludges, large amounts of Ni; and Stratford sludge, a large amount of Mo. Several of the sludges contained large amounts of Zn, Cu, Pb and Cr, but they all showed normal amounts of Mn, As and Hg.

PCB concentrations in the sludges (Table 2) ranged from 0.13 to 1.61 µg/g dry solids and were approximately an order of magnitude smaller than values reported by Environment Canada (1976) and Shannon et al (1976). They were also smaller than values calculated from Lawrence and Tosine (1977). The

low values observed in this study possibly reflect the 1971 legislation restricting industrial uses of PCB's.

3.3 Sludge Constituent Loadings to the Application Sites

Sludge constituent loadings to the application sites, estimated with data from Tables 1 and 2 are presented in Table 3. They varied widely from site to site and frequently exceeded the average contents in uncontaminated Ontario soils. Cadmium addition to the Burlington site sampled in 1976; Zn and Cr additions at Kitchener; and Ni and Mo additions at Stratford greatly exceeded the maximum recommended loadings to Ontario agricultural lands. Cadmium addition to the Galt, Guelph, Oakville and Stratford sites; Zn addition to the Burlington, 1976 and Oakville sites; and Pb addition to the Burlington, 1976 site exceeded the maximum recommended loadings. The PCB loadings to all sites were small.

3.4 Effects of Sludge Application on Metals and PCB's in Soils

The total metal and PCB contents of soils (Table 4) and the 0.005 M DTPA-extractable metal contents of soils (Table 5) are averages of four replicates except for the 1978 Oshawa control values which are averages of two replicates. The 1978 Oshawa data were not included in the statistical analyses.

Analysis of variance indicated that the total metal and PCB contents of replicate soil samples were not significantly different by F test. However, differences between locations and sludge treatments were significant at the 0.01 probability level. Covariance analysis indicated that the metal and PCB contents of the sludge treated soils were related to sludge addition and not to the initial contents of these constituents in the soils.

The metal and PCB contents of composite and grab soil samples taken as described in Section 2.3 were not significantly different (Tables 6 and 7). The standard errors for comparable means were approximately equal indicating that the two sampling techniques resulted in data with the same degree of variability. It was concluded that composite soil sampling offered no advantage over grab sampling for either the control or sludge treated soils.

TABLE 3. A COMPARISON OF ESTIMATED SLUDGE CONSTITUENT LOADINGS TO THE APPLICATION SITES WITH THE AVERAGE CONTENTS IN UNCONTAMINATED ONTARIO SOILS AND RECOMMENDED MAXIMUM LOADINGS

Site		Dry Solids (tonnes/ha)	Cd	Zn	Cu	Ni	Pb	Cr	Mn	Mo	As	Hg	PCB's
							Estimated Loadings (kg/ha)						
Brantford		54	1.2	119	67	11	66	38	13	<1.1	0.9	0.17	0.028
Burlington	1976	90	19	455	122	36	112	160	27	<1.8	3.2	0.36	0.145
	1978	9	1.9	45	12	3.6	11	16	2.7	<0.2	0.3	0.04	0.015
Galt		45	2.4	67	53	2.4	14	57	12	<0.9	0.4	0.25	0.007
Georgetown		18	0.8	12	10	0.9	21	5.7	3.3	<0.4	0.1	0.04	0.005
Guelph	1976	18	3.7	130	66	1.6	26	70	4.9	1.2	0.2	0.28	0.013
	1978	9	1.8	65	33	0.8	13	35	2.4	0.6	0.1	0.14	0.006
Kingston	1976	36	0.3	64	22	0.9	32	50	9.7	1.2	0.3	0.38	0.030
	1978	Large	-	-	-	-	-	-	-	-	-	-	-
Kitchener		90	1.1	595	59	30	55	628	23	<1.8	0.9	0.28	0.058
Oakville	1976	61	2.7	416	92	21	82	123	17	<1.2	0.4	0.49	0.024
	1978	68	3.0	463	102	23	91	137	19	<1.3	0.4	0.54	0.027
Oshawa		10	0.1	61	6.8	11	13	66	3.2	0.6	0.2	0.03	<0.001
Stratford		94	3.1	258	111	118	90	145	44	19.4	1.7	0.32	0.021
							Average Contents in Uncontaminated Ontario Soils* (kg/ha)						
		-	1.6	110	50	32	30	30	-	4	14	0.2	-
							Recommended Maximum Loadings to Ontario Soils* (kg/ha)						
		-	1.6	330	150	32	90	210	-	4	14	0.8	-

* Values obtained from the "Guidelines for Sewage Sludge Utilization on Agricultural Lands", October, 1980. Prepared by an ad hoc committee of the Ontario Ministry of Agriculture and Food and the Ontario Ministry of the Environment.

TABLE 4. TOTAL METALS AND PCB's (µg/g) IN THE SOILS[1].

Location	Treatment	Cd 1978	Zn 1978	Cu 1978	Ni 1978	Pb 1978	Mo 1978	As 1976	Hg 1976	PCB 1976	PCB 1978
Brantford	Control	1.06	222	31.6	18.6	33.0	NM	7.2	0.11	ND	0.025
	Sludge	1.19	164*	30.9	18.5	28.9	NM	4.8*	0.08	0.004	0.023
Burlington	Control	0.54	119	28.1	23.6	26.0	NM	5.2	0.04	0.003	0.013
	Sludge	0.92	107	26.5	24.4	26.8	NM	5.1	0.05	<0.003	0.022
Galt	Control	0.26	101	17.0	14.8	25.6	NM	4.2	0.04	<0.003	0.007
	Sludge	1.14*	130	27.9	17.6	30.1	NM	3.7	0.15*	0.030	0.067
Georgetown	Control	0.54	71	32.2	20.0	21.2	NM	2.7	0.04	0.018	0.020
	Sludge	0.89	97	40.9	24.4	27.5	NM	4.1*	0.04	0.003	0.018
Guelph	Control	0.45	120	15.5	14.1	24.9	NM	3.3	0.03	<0.003	0.012
	Sludge	1.94*	383*	58.4*	14.1	42.9*	NM	3.6	0.08	0.008	0.215*
Kingston	Control	0.44	97	15.5	21.4	21.5	NM	1.1	0.03	<0.003	0.008
	Sludge	0.70	91	19.9	11.6*	27.0	NM	1.5	0.20*	0.125	0.220*
Kitchener	Control	0.41	88	14.0	13.0	24.0	NM	4.2	0.06	ND	0.009
	Sludge	0.89	148*	37.5*	21.4*	29.0	NM	6.3*	0.13*	0.050	0.192*
Oakville	Control	0.26	104	19.8	23.2	26.6	NM	6.1	0.04	ND	0.015
	Sludge	1.13*	257*	62.9*	31.0*	36.0*	NM	6.6	0.10*	0.099	0.453*
Oshawa	Control	0.13	126	25.5	25.2	27.0	NM	2.1	0.06	0.019	0.015
	Sludge	0.62	394	62.2	77.0	96.1	NM	4.7*	0.13*	0.037	0.111
Stratford	Control	0.32	98	21.6	22.5	23.8	ND	5.9	0.09	ND	0.008
	Sludge	1.23*	131	44.4*	32.5*	34.0*	19.2	5.4	0.16*	0.057	0.103*
Mean	Control	0.44	115	22.1	19.6	25.4	-	4.2	0.05	-	0.013
	Sludge	1.06	190	41.1	27.2	37.8		4.6	0.11	-	0.142

1 The data are averages of four replicates except for 1978 Oshawa control where they are averages of two replicates. Mean values include the Oshawa 1978 data, but the Duncan's Multiple Range Test did not include those data.

* Indicates that the sludge and control values are significantly different at the 0.05 probability level.

NM Not measured.

ND Not detected.

TABLE 5. 0.005 M DTPA-EXTRACTABLE METALS (μg/g) IN THE 1978 SOILS[1]

Location	Treatment	Cd	Zn	Cu	Ni	Pb	Mo
Brantford	Control	0.30	9.6	3.5	0.49	3.33	NM
	Sludge	0.36	12.1	5.2	0.54	2.94	NM
Burlington	Control	0.12	1.7	1.1	0.35	1.98	NM
	Sludge	0.23	2.5	1.5	0.36	2.08	NM
Galt	Control	0.10	1.6	0.5	0.13	1.52	NM
	Sludge	0.51*	8.8	4.7	0.73	2.25	NM
Georgetown	Control	0.14	1.5	2.8	0.77	2.51	NM
	Sludge	0.24	4.5	3.8	0.78	3.12	NM
Guelph	Control	0.12	1.5	0.5	0.13	1.84	NM
	Sludge	1.00*	65.1*	18.8*	0.44	4.80*	NM
Kingston	Control	0.09	1.6	0.6	0.37	1.01	NM
	Sludge	0.13	4.9	3.8	0.18	3.92*	NM
Kitchener	Control	0.10	1.4	0.5	0.13	1.60	NM
	Sludge	0.41*	22.6*	9.9*	2.21*	2.66	NM
Oakville	Control	0.09	0.7	0.6	0.42	2.07	NM
	Sludge	0.42*	40.2*	18.3*	2.72*	5.66*	NM
Oshawa	Control	0.18	7.2	2.0	0.76	2.92	NM
	Sludge	0.28	64.7	16.0	9.84	22.3	NM
Stratford	Control	0.13	1.4	1.0	0.39	2.39	ND
	Sludge	0.45*	12.5	8.4*	2.84*	3.85	1.42
Mean	Control	0.14	2.8	1.3	0.39	2.12	-
	Sludge	0.40	23.8	9.0	2.06	5.36	-

1 The data are averages of four replicates except for Oshawa control where they are averages of two replicates. Mean values include the Oshawa data, but the Duncan's Multiple Range Test did not include those data.

* Indicates that the sludge and control values are significantly different at the 0.05 probability level.

NM Not measured.

ND Not detected.

TABLE 6. COMPARISON OF TOTAL METALS AND PCB's (μg/g) IN COMPOSITE AND GRAB SOIL SAMPLES TAKEN IN 1978

Location	Sample	Cd	Zn	Cu	Ni	Pb	PCB
				Control			
Guelph	Composite	0.45±0.19*	120±5	15.5±1.3	14.1±1.8	24.9±2.1	0.012±0.002
	Grab	0.37±0.10	116±7	14.8±0.9	14.8±0.9	28.4±1.5	0.008±0.003
Kitchener	Composite	0.41±0.09	88±9	14.0±0.6	13.0±0.8	24.0±1.1	0.009±0.008
	Grab	0.53±0.07	89±3	14.9±0.8	14.4±0.4	22.5±0.8	0.117±0.002
				Sludge			
Oakville	Composite	1.13±0.37	257±26	63±8	32±1	36±4	0.452±0.058
	Grab	1.22±0.12	252±19	64±5	31±1	34±3	0.372±0.032
Oshawa	Composite	0.62±0.16	394±46	62±6	77±5	96±10	0.106±0.036
	Grab	0.74±0.10	417±28	64±4	82±6	110±11	0.103±0.014

* Mean and standard error; n=4 and 6 for composite and grab samples, respectively.

TABLE 7. COMPARISON OF DTPA-EXTRACTABLE METALS (μg/g) IN COMPOSITE AND GRAB SOIL SAMPLES TAKEN IN 1978

Location	Samples	Cd	Zn	Cu	Pb
		Control			
Guelph	Composite	0.12±0.01*	1.49±0.23	0.48±0.05	1.84±0.10
	Grab	0.12±0.02	1.46±0.25	0.34±0.11	1.82±0.22
Kitchener	Composite	0.11±0.01	1.37±0.06	0.45±0.06	1.60±0.07
	Grab	0.10±0.01	1.23±0.07	0.47±0.06	1.89±0.36
		Sludge			
Oakville	Composite	0.42±0.06	40±8	18.3±4.8	5.66±0.78
	Grab	0.42±0.04	37±4	19.1±2.7	5.72±0.83
Oshawa	Composite	0.28±0.03	65±7	16.0±1.6	22.3±1.1
	Grab	0.30±0.02	81±6	18.1±1.4	21.9±1.4

* Mean and standard error; n=4 and 6 for composite and grab samples, respectively.

3.4.1 Total metals and PCB's

Measurements of total Cd, Zn, Cu, Ni, Pb, Cr and Mo for the 1976 soil samples exhibited large variations that were attributed to analytical error. However, it was impossible to repeat the analysis of 1976 soils because the samples had been discarded. The sampling and analysis was repeated in 1978 and, except for Cr, the measurements were highly reproducible. Cadmium, Zn, Cu, Ni, Pb, Mo and PCB data for the 1978 soils and As, Hg and PCB data for the 1976 soils are presented in Table 4. No values are reported for Cr; however, studies are in progress to resolve the analytical difficulties with that metal. PCB contents measured in the 1978 soil samples were larger than those measured in 1976 samples. The increases with time in treated soils might be attributed to sludge addition, however, this explanation would not account for increases in the control soils. It is probable that the differences from 1976 to 1978 were largely related to small differences in analytical technique. The 1976 measurements were done at Guelph, and the 1978 measurements were done at Burlington.

In general, sludge treatment increased the total metal and PCB contents of soils. Mean values for the treated soils were much larger than for the control soils. The largest increases observed at Guelph, Kitchener, Oakville, Oshawa and Stratford were in general agreement with estimated sludge constituent loadings (Table 3). Soil measurements reflected a very large Mo loading at Stratford but did not reflect very large loadings of Zn at Kitchener or Ni at Stratford. At several locations Zn exhibited large increases and Cu, Pb, Ni, Cd, Hg and PCB's exhibited smaller increases in that order. There was no consistent effect of sludge treatment on the As contents of soils. Data for the Brantford location were not consistent with the observation that sludge treatment increased the metal contents of soils. It was concluded that the Brantford control soil probably had received sludge because the total metal contents generally; (a) exceeded values for the Brantford sludge treated soil, (b) exceeded the mean values for all control soils, and (c) approximated the mean values for all sludge treated soils.

Cadmium, Cu, Ni, As and Hg contents of the control soils approximated mean values of 0.8, 25, 16, 7 and 0.1 $\mu g/g$, respectively, reported for uncontaminated Ontario soils (OMAF/OMOE, 1980). Zinc and Pb contents of the control

soils exceeded the 55 and 15 μg/g values reported for uncontaminated Ontario soils but fell within the 5 to 300 μg Zn/g and 5 to 71 μg Pb/g ranges reported as background levels in Canadian soils (McKeague et al, 1979). The metal contents of sludge treated soils occasionally exceeded, but generally were smaller than the recommended maximum values of: Cd, 1.6; Zn, 220; Cu, 100; Ni, 32; Pb, 60; As, 14; and Hg, 0.5 μg/g for Ontario soils (OMAF/OMOE, 1980). The Mo content of soil treated with Stratford sludge greatly exceeded the 4 μg/g recommended maximum value. Increased metal contents in soils attributed to sludge treatment were consistent with reports from the Regional Municipality of Halton in Ontario (OMOE, 1977), the United States (Chaney et al, 1977) and the United Kingdom (Berrow and Webber, 1972).

Generally, higher levels of PCB's in sludge treated than in control soils (Table 4) indicated a buildup due to sludge application on land. Environment Canada (1976) reported 0.005 μg PCB/g in two southern Ontario soils which had not received sludge and a range from "not detected" to 0.715 μg/g in twenty-five soils which had received one or more applications of sludge.

3.4.2 DTPA-extractable metals

The 0.005 M DTPA-extractable metals in soils were highly correlated with the total metals. The correlation coefficients were: Cd, 0.74; Zn, 0.86; Cu, 0.91; Ni, 0.70; and Pb, 0.77. Sludge treatment increased the levels of DTPA-extractable Cd, Zn, Cu, Ni and Pb and mean values for the treated soils were much larger than for controls (Table 5). Similar increases due to land application of sludge in the United States have been reported by Chaney et al (1977). In the present study, the largest increases were observed at Guelph, Kitchener, Oakville, Oshawa and Stratford and coincided with large increases of total metals (Table 4). Molybdenum was detected in the sludge treated soil at Stratford but not in the control. Chromium was not detected in any soil.

3.5 Effects of Sludge Application on the Total Metal and PCB Contents of Plant Materials

The total metal contents reported for 1976 and 1978 plant materials and the total PCB contents for 1976 plant materials (Tables 8 and 9) are

TABLE 8. TOTAL METALS AND PCB's (μg/g) IN THE 1976 PLANT MATERIALS[1]

Location	Crop	Treatment	Cd	Zn	Cu	Ni	Pb	Mo	Mn	Co	Cr	As	Hg	PCB
Brantford	Corn	Control	0.07	47	10.6	1.68	0.4	0.7	51	1.9	3.0	0.02	0.010	0.031
		Sludge	0.09	50	11.1	1.74	0.9	0.8	43	1.2	3.1	0.01	0.012	0.041
Burlington	Oats	Control	0.11	14	4.5	3.99	0.6	1.4	43	2.6	7.4	0.06	0.021	0.034
		Sludge	0.21	20	5.9	4.70	0.6	0.9	81	2.8	9.2	0.07	0.016	0.048
Galt	Corn	Control	0.08	26	10.2	1.45	0.5	0.5	39	2.1	2.4	0.01	0.011	0.046
		Sludge	0.18	32	9.8	1.53	0.9	0.6	42	2.6	2.4	0.01	0.010	0.043
Georgetown	Corn	Control	0.22	23	11.6*	1.10	0.4	1.2	38*	2.1	1.6	0.02	0.023	0.023
		Sludge	0.15	24	13.9	1.18	0.6	0.8	54	2.2	1.4	0.01	0.024	0.010
Guelph	Corn	Control	0.14*	24	7.0*	2.98	0.4	1.0	59*	2.4	4.1*	0.01*	0.017	NM
		Sludge	0.74	28	10.4	4.22	0.5	1.6	37	2.2	10.0	0.04	0.014	NM
Kingston	Corn	Control	0.13	29	8.0	1.15	1.1	0.8	35	2.9	2.6	0.01	0.016*	0.025
		Sludge	0.08	38	7.1	1.66	0.7	1.5	25	1.8	2.4	0.01	0.026	0.022
Kitchener	Corn	Control	0.05	24*	8.2*	1.46	0.5	0.6	38	1.8	1.8	0.01*	0.018	0.043
		Sludge	0.15	38	11.9	1.49	0.8	1.6	45	2.2	2.2	0.03	0.023	0.030
Oakville	Wheat	Control	0.13	8.8	1.6	3.05	0.6	0.4	64	2.0	6.3	0.01	0.019	0.019
		Sludge	0.32	110	2.2	3.90	0.6	0.7	31	2.4	6.1	0.04	0.032	0.013
Oshawa	Oats	Control	0.08	54	6.8	11.0	1.0	1.6	36	2.6	17.9	0.16	0.034	0.055
		Sludge	0.08	29	5.4	5.27	0.9	1.8	20	3.3	10.2	0.07	0.041	0.035
Stratford	Corn	Control	0.12	23	7.0	2.19	0.4	1.1*	58*	2.2	3.2	0.04	0.013	0.036
		Sludge	0.07	32	7.9	2.12	0.4	23.7	38	2.1	2.7	0.04	0.019	0.033
Mean		Control	0.11	27	7.6	3.01	0.6	0.9	46	2.3	5.0	0.03	0.018	0.035
		Sludge	0.21	40	8.6	2.78	0.7	3.4	42	2.4	5.0	0.03	0.022	0.030

1 Corn leaves and oat and wheat leaves and stems were analyzed. The data are averages of four replicates. The mean values include all data but the Duncan's Multiple Range Test included just the corn data.

* Indicates that the sludge and control values are significantly different at the 0.05 probability level.

NM Not measured

TABLE 9. TOTAL METALS AND PCB's (μg/g) IN THE 1978 PLANT MATERIALS*

Location	Crop	Treatment	Cd	ZN	Cu	Ni	Pb	Mo	Cr	PCB
Brantford	Corn	Control	0.10	57	9.5	0.35	1.99	0.68	0.52	NM
		Sludge	0.10	62	9.5	0.48	1.29	0.62	0.42	NM
Burlington	Oats	Control	0.10	26	4.9	0.62	0.73	1.22	0.48	0.026
		Sludge	0.18	33	5.5	1.08	0.68	0.72	0.92	NM
Galt	Corn	Control	0.08	45	9.4*	0.55	1.71	0.68	0.48	NM
		Sludge	0.10	36	6.9	0.50	1.11	0.58	0.40	NM
Georgetown	Corn	Control	0.11	33	9.0	0.60	1.24	0.82	0.42	NM
		Sludge	0.10	34	9.7	0.52	1.44	0.55	0.40	NM
Guelph	Corn	Control	0.10*	40*	9.7	0.52	1.26	0.70	0.38	0.036
		Sludge	0.20	122	9.5	0.45	1.93	0.80	0.40	0.053
Kingston	Corn	Control	0.13	38	6.9	0.52	1.86	0.40	0.38	NM
		Sludge	NS	NS	NS	NS	NS	NS	NS	NS
Kitchener	Corn	Control	0.13	40*	7.2	0.62	2.22	0.80	0.78	0.033
		Sludge	0.11	73	6.0	0.62	2.25	0.90	0.42	0.027
Oakville	Corn	Control	NS	NS	NS	NS	NS	NS	NS	NS
		Sludge	0.22	66	11.4	0.52	1.95	0.50	1.38	0.052
Oshawa	Corn	Control	NS	NS	NS	NS	NS	NS	NS	NS
		Sludge	0.13	55	8.5	0.52	2.27	0.70	0.48	0.036
Stratford	Corn	Control	0.16	30	8.0	0.45	0.97	0.72*	0.55	NM
		Sludge	0.14	41	7.8	0.35	1.25	32.2	0.52	NM
Mean		Control	0.11	39	8.1	0.53	1.50	0.75	0.50	-
		Sludge	0.14	58	8.3	0.56	1.57	4.17	0.59	-

1 Corn leaves and complete oat plants were analyzed. The metal data are averages of four replicates and the PCB data are averages of four or fewer replicates. Mean values include all data but the Duncan's Multiple Range Test included just the corn data from Brantford, Galt, Georgetown, Guelph, Kitchener and Stratford.

* Indicates that the sludge and control samples are significantly different at the 0.05 probability level.

NM Not measured.

NS No sample.

averages of four observations. The total PCB contents for 1978 plant materials are averages of four or fewer observations. Four replicate plant material samples were obtained from all locations except the 1978 Kingston sludge site and the Oakville and Oshawa control sites. Statistical analyses were conducted with all of the metal and PCB data for corn sampled in 1976 and with the metal data for corn sampled from Brantford, Galt, Georgetown, Guelph, Kitchener and Stratford in 1978.

Analysis of variance indicated no significant differences in the metal and PCB contents of replicate corn leaf samples. Furthermore, Pb and Co in the 1976 samples and Cr in the 1978 samples exhibited no significant differences between locations and treatments. The remaining data exhibited significant differences between locations and sometimes between treatments. Covariance analysis indicated that Ni in the 1976 and 1978 samples and Cu in the 1978 samples were related to the initial contents of these metals in the soils as well as to sludge treatment.

The metal and PCB contents of composite and grab plant material samples taken as described in Section 2.4 were not significantly different (Table 10). The standard errors for comparable means were approximately equal indicating that the two sampling techniques resulted in data with a similar degree of variability. It was concluded that composite sampling offered no advantage over grab sampling for plant materials grown on either the control or sludge treated sites.

The Cd, Zn, Cu, Mo and PCB contents of 1976 and 1978 plant materials were similar (Tables 8 and 9). However, the Ni and Cr contents of 1976 plant materials were larger and the Pb contents of 1976 plant materials were smaller than for 1978 plant materials. The authors are unable to explain these differences but suspect analytical error and place more confidence in the 1978 data. Analyses were obtained for wheat, oat and corn plant materials but there were too few wheat and oat data to permit a comparison of measurements for the different crops.

In general, sludge treatment exhibited small effects on plant composition. The differences between locations frequently were larger than the sludge effects within locations. The most consistent effects were to increase the Cd and Zn contents of 1976 plant materials and to increase the Zn contents

TABLE 10. COMPARISON OF TOTAL METALS (μg/g) IN COMPOSITE AND GRAB PLANT MATERIAL SAMPLES TAKEN IN 1978

Location	Sample	CD	Zn	Cu	Ni	Pb	Cr	Mo
				Control				
Guelph	Composite	0.10±0.01*	40±2	9.7±0.2	0.52±0.05	1.26±0.15	0.38±0.05	0.70±0.04
	Grab	0.11±0.02	40±2	9.8±0.4	0.52±0.05	1.29±0.11	0.48±0.11	0.60±0.10
Kitchener	Composite	0.13±0.02	40±2	7.2±0.4	0.62±0.19	2.22±0.08	0.78±0.30	0.80±0.04
	Grab	0.10±0.02	36±1	7.1±0.5	0.45±0.03	1.89±0.10	0.40±0.05	0.78±0.07
				Sludge				
Oakville	Composite	0.22±0.02	66±8	11.4±0.5	0.52±0.08	1.95±0.15	1.38±0.19	0.50±0.06
	Grab	0.12±0.01	48±2	11.1±0.4	0.77±0.14	1.55±0.13	1.00±0.13	0.55±0.08
Oshawa	Composite	0.13±0.02	55±2	8.5±0.4	0.52±0.02	2.27±0.17	0.48±0.08	0.70±0.11
	Grab	0.15±0.02	61±6	8.6±0.7	0.58±0.06	2.08±0.24	0.48±0.10	0.60±0.07

* Mean and standard error; n=4 and 6 for composite and grab samples, respectively.

of 1978 plant materials. However, these increases were generally small and not well related to the estimated metal loadings in sludge (Table 3) or to total metals (Table 4) or DTPA-extractable metals (Table 5) in the soils. There was no consistent effect of sludge treatment on the Cu, Ni, Pb, Mo (except at Stratford), Mn, Cr, As, Hg and PCB contents of plant materials. Stratford sludge contributed a heavy Mo loading to soil and greatly increased the Mo content of corn leaves.

Plant materials grown on the sludge application sites generally exhibited metal contents within the normal ranges defined by Blakeslee (1976) for agricultural crops as follows: Cd, 0.05 to 0.20; Zn, 15 to 150; Cu, 3 to 40; Ni, 0.1 to 1.0; Pb, 0.5 to 5.0; Mo, 0.2 to 1.0; Mn, 15 to 150; Cr, 0.1 to 0.5; As, 0.01 to 1.0; and Hg, 0.001 to 0.01 μg/g. Corn leaves from the Stratford sludge utilization site greatly exceeded the normal Mo contents and would be expected to cause molybdenosis (molybdenum-induced copper deficiency) in ruminant animals. Dietary levels of 10 to 20 μg Mo/g are nearly always associated with some evidence of disturbed copper metabolism in ruminants (Allaway, 1968). The Cd contents of plant materials increased at several of the sludge application sites and exceeded the normal range at the Guelph and Oakville locations in 1976 (Table 8). Increased levels of Cd are of concern because it has no known biologic function and is toxic to both plants and animals. It accumulates in the liver and kidneys of animals and has been linked with a number of human ailments including renal dysfunction, hypertension and cardiovascular disease. Safe levels of Cd have not been defined for food or feedstuffs, however, it is unlikely that levels up to 1 μg Cd/g in feedstuffs present a hazard to human health. Increased levels of Zn in plant materials resulting from sludge application are desirable because there is widespread concern over deficiency for both plants and animals (Allaway, 1968). The danger of toxicity from excess dietary Zn is minimal and plant toxicity occurs at concentrations in plant materials exceeding approximately 500 μg/g.

Plant materials grown on the sludge application sites exhibited no tendency to accumulate PCB's. The PCB contents observed were low and were in good agreement with previous reports for sludge application sites in Ontario (Environment Canada, 1976 and Lawrence and Tosine, 1977).

3.6 Crop Quality in Relation to the Ontario Sludge Utilization Guidelines

The results of this study indicated that sludge application to land according to the OMAF/OMOE (1980) Guidelines will not cause deleterious uptake of heavy metals by corn, oats and wheat. Much increased concentrations of Mo, Cd and Zn in the crops were observed only where metal additions to soil greatly exceeded the maximum recommended loadings. It was evident that the Guidelines allow an appreciable safety margin which is considered desirable because sludge composition may vary widely over time (Monteith and Stephenson, 1978).

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